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In re application of

Jean-Paul SALOME et al. Conf. 8466

Application No. 10/666,223 Group 1653

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PROCESS FOR EXTRACTING THE COMPONENTS OF PEA FLOUR

DECLARATION UNDER RULE 132

Assistant Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Jean-Marc VERRIN hereby declare:

I am an expert in starch manufacturing processes, including processes for extracting and refining starch.

The claimed invention relates to a process for extracting and refining the components of pea flour with the aid of at least one of the pieces of equipment borrowed from a potato starch factory.

The manufacturing processes and equipment lines for starch are specifically designed for each type of starch (e.g., wheat, corn, or potato). Thus, the machinery and equipment used to produce each type of starch is specific to that starch.

This is evidenced by the attached two publications, namely: K. Rausch, *Front End to Backpipe: Membrane Technology in*

the Starch Processing Industry that teach that the processing of raw materials (e.g., maize, pea, potato, and corn) and an excerpt from the website of the French Association of Starch Manufacturers (translation also provided)

As each type of starch requires specific equipment and a particular process, this leads to an inefficient use of the manufacturing site. Because the process and industrial tools at the manufacturing site are dedicated to a particular type of starch, the manufacturing site is not capable of being used throughout the year. Rather, the manufacturing site is used only when the raw material for the particular type of starch is available.

The present invention extracts and refines the components of pea flour, i.e., the starch, the proteins, the internal fibers and the solubles, with the aid of of equipment borrowed from a potato starch factory. In particular, the present invention makes it possible to extract and refine these components without the need to remove beforehand the internal fiber components of the pea. By doing so, the pea flour can use a similar process with the same type of equipment that is used to produce potato starch.

In my opinion, the proposed combination of patents relied upon in the Official Action of March 31, 2006 does not suggest such a process.

The undersigned declare further that all statements made herein of their own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Jean-Marc VERRIN

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Front End to Backpipe: Membrane Technology in the Starch Processing Industry

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The starch processing industry is characterized by streams that vary in diversity and complexity and that require extensive processing to achieve high end product quality. Water removal and product separations are two fundamental processing steps that impact product quality and processing economics. Many uses have been found for membrane technology in the starch processing industry; these include pretreatment of fresh water, recovery of solids and wastewater treatment. In many cases, membrane applications have increased quality of products, decreased energy costs and reduced disposal issues relating to waste treatment. Membranes can be used to filter many types of fluids in potato, wheat and corn starch isolation processes with varying degrees of success. Robust membrane materials have been developed with unique processing capabilities suited for the needs of the starch processing industry. Membranes have been used commercially to increase product quality while reducing costs, such as in syrup and sweetener clarification. Membrane technology has shown promise for reducing evaporation costs, improving product recovery and removing solids prior to wastewater treatment. In many applications, the cost of dewatering using membranes was a fraction of the cost of traditional methods. Membranes can be used to recover proteins from dilute process streams, but more work is needed regarding changes in nutrient quality when membranes are used in place of conventional separation technologies such as centrifugation, vacuum belt filtration and evaporation. There is need for detailed discussion of analyses regarding economics of long term operation and maintenance of membranes as part of a processing system.

Keywords: Membrane filtration; Starch processing

1 Introduction

Membrane technology is a method to recover processing solids and treat water for recycling in many food processing industries. Several reviews have been published that describe how membrane technology has been applied to grain processing, including the starch processing industry [1, 2]. These provide a general overview of previous research. A comprehensive review of membrane applications in the starch processing industry should be helpful because membrane technology is changing rapidly and the starch industry is facing new challenges. Membranes are becoming more robust and allowing more applications in severe environments. Membrane materials are improving rapidly; applications found to be marginal or unfavorable toward membrane application a decade ago can now be economically feasible. Furthermore, the starch processing industry has ever increasing needs for higher value and maximum yields from every facility, due to more restrictive environmental regulations and increasing landfill and waste treatment costs. Membrane technology will help provide solutions for many of these challenges.

Application of membrane technology has several advantages for the starch processing industry. Costs of removing water to concentrate starch and coproducts can be reduced. Up to 90% savings in energy have been reported using membranes compared to evaporation. Membranes have the ability to achieve separations without application of heat to the product, improving coproduct quality [1]. Reduction of exposure to heat is conventionally thought to improve nutritional quality and digestibility of proteins. However, few publications have studied the effects of membrane technology on nutritional quality when heat exposure has been reduced [3]. Membranes can be used to allow recovery and recirculation of water used in processes, reducing water needs. Some process streams from membrane filtration may be more efficiently handled by evaporators due to reduced surface fouling of heated evaporator surfaces [4]. New coproducts could be developed using membrane technology. Waste treatment costs could be reduced by using membranes to recover solids

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Review

from a process stream that would otherwise enter the wastewater treatment facility. This results in twofold savings for the processor, as costs could be reduced in waste treatment as well as more solids could be recovered in coproducts that can be marketed.

There are disadvantages with membrane technology that must be addressed. Some new membrane processes must be documented for food safety by regulatory agencies, resulting in increased costs and delays in bringing the new process into production. Even though lab and pilot scale studies may have been conducted with a new application, there can be uncertainty about membrane durability, length of operating life and replacement costs. Some membrane designs have limits on operating pressures that prevent implementation in certain processes. Certain feed stocks in cereal processing can have high fouling rates that prevent economical use of membranes [1].

Use of membranes can also mean dealing with changes in corporate culture, as membrane technologies can be perceived as being more difficult to operate and more expensive and trouble prone. Overcoming these (nontechnical) disadvantages can be difficult due to lack of objective data suited for commercial end use. Research conducted two decades ago focused on ability of a particular membrane to filter out certain constituents of interest to the starch industry. Attention was directed toward preliminary economic feasibility to determine energy savings and product quality. Presently, research is needed to determine long term cleaning strategies on economic feasibility of particular membrane applications. This review will show that adaptation of membrane technology to starch processing has progressed through several stages. Initially, membrane manufacturers produced materials that had various pore sizes and chemical resistances. Manufacturers developed materials for microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO), initially thought to be suitable for starch processing. Eventually, membranes became more effective in the starch process industry. Research has shown that newer membranes could make efficient and precise separations that did not require large amounts of energy. Experiments were typically on a small scale and often used model process materials to investigate whether membrane materials would have any benefit to the processor. Researchers and membrane companies developed membranes that could recover solids, fractionate nutrients and generate clean water that could be reused within the process. This ability would allow processors to recover more solids from process streams, create new coproducts and reduce the burden on the environment and wastewater treatment.

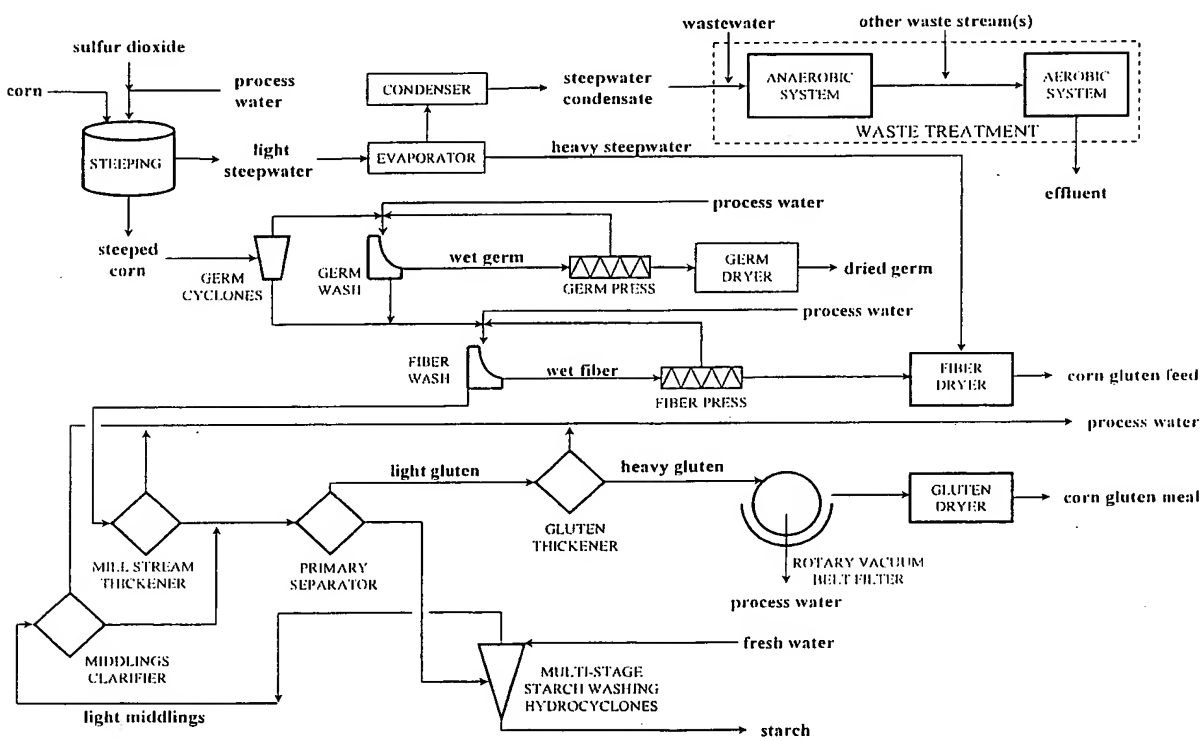

Fig. 1. Schematic of the corn wet milling process used for starch production from corn [5, 6].

2 Corn Starch Processing

The wet milling process begins by soaking corn in a solution of weak sulfurous acid for 24 to 48 h in a semicontinuous steeping system that hydrates and softens the kernel and leaches solubles from the germ (Fig. 1). Wet milling process results in coproducts containing concentrated amounts of starch, protein, fiber and oil. Corn wet milling is a relatively sophisticated process, with large capital investment and a large economy of scale [5, 6]. Typical plants in the US process at least 2,500 t of corn per day and operate nearly 365 days per year. Historically, wet milling has provided pure (>99.5%) starch products for the paper and corrugating industries, modified starches for food ingredients and high fructose corn syrup (HFCS). By the late 1970s, the wet milling industry was growing at a rapid pace to supply HFCS to the beverage market. While the HFCS market has matured, demand for fuel ethanol has grown, continuing an increase in production capacity.

Corn wet milling plants generate large amounts of wastewater, although the process removes a large portion of process water in steepwater evaporators and dryers for germ, corn gluten feed and corn gluten meal. *Ray* et al. [7] reported that steepwater had a high waste load, exceeding a chemical oxygen demand (COD) of 200,000 mg/L and preventing direct discharge from a facility. *May* [6] reported that the wet milling process generated 1,300 to 1,600 L of light steepwater (LSW) per t of corn. For example, a corn wet mill that processed 6,300 t per day sent over 3.8×10^6 L of LSW to steepwater evaporators every day. Fresh water enters the plant near the end of the process at the starch washing step, flows countercurrent to corn solids in the process and exits with steepwater solids or coproducts. Water from steeping is removed by steepwater evaporators or by gluten feed dryers. Typically, 2.1 to 2.5 kg water per kg dry starch is used to remove residual protein from starch using multistage hydrocyclone systems. Since water introduced into the process is removed by evaporation, the amount of water used to wash starch is linked to evaporative capacity of the plant.

Insoluble materials in LSW could originate from broken corn, bacterial cells or material brought in with process water used to make fresh steepwater. Following steeping, LSW may be centrifuged to remove suspended solids and used as a nutrient source for fermentation during ethanol production. A conventional corn wet milling process will concentrate LSW to 45 to 50% total solids (TS) using a multiple effect evaporator or mechanical vapor recompression [5]. Protein and other components in LSW tend to adhere to heat exchanger surfaces, causing fouling of the evaporators and loss of plant capacity and increased maintenance, labor and capital costs.

In the early 1980s, *Cicuttini* [8, 9], *Cicuttini* et al. [10] and *Kollacks* and *Rekers* [11] demonstrated that membrane technology had progressed sufficiently to withstand rigors of commercial corn processing. *Cicuttini* [8, 9] patented a concept in which RO membranes were installed to remove protein and other solids from the light middlings stream (Fig. 1). Solids in this stream flow from starch washing back to the primary starch separator or clarifier. The light middlings stream contained starch and gluten particles in suspension with 10,000 mg/L soluble protein. *Cicuttini* et al. [10] reported data from a small scale study in which a membrane having 0.78 m^2 area was used to perform RO filtration. The study used membranes made from cellulose acetate arranged in a tubular configuration, each tube having 14.4 mm inside diameter and 6 m length. Batches of light middlings were passed through the RO system, with concentrate returned to the batch tank. It was found that a crossflow velocity inside the membrane tubes of 1.75 to 2.5 m/s gave similar filtration results. Membranes could operate up to 40 h without any reduction in permeate flux rates due to fouling. These tests were followed up with a pilot scale experiment using the same type of membranes at a commercial wet milling facility. This system used tubular RO membranes with 11.4 m^2 area. The tests found that fouling was not apparent up to 70 h of operation and protein levels were reduced to 44 to 71 mg/L. Due to promising results at a smaller scale, a commercial scale system was subsequently started. The commercial system used a cellulose acetate construction with 141 membrane modules and contained 268 m^2 of membrane area. This system operated at a corn wet milling facility, at a crossflow velocity of 2.5 m/s and a maximum of 4,000 kPa pressure before cleaning. The circulation pump supplied 15 m^3/h to the membrane filtration stage. The system produced approximately 5 m^3/h of permeate containing 100 to 140 mg/L protein. Membranes were cleaned every 4 to 5 days with an enzymatic agent; cleaning costs were reported at $ 0.08 /m^3 of permeate. No irreversible fouling of the membranes was observed.

Kollacks and *Rekers* [11] followed up the work of *Cicuttini* et al. [10]; this included five years of data collection with a commercial scale RO system designed to filter the light middlings stream in a plant processing 300 t per day. The unit used on the light middlings stream had a single stage with tubular cellulose acetate membranes having 399 m^2 total membrane area arranged in a feed and bleed configuration. Each bundle of membranes, or module, consisted of seven tubes, each being 14.4 mm in diameter and 6 m in length. RO improved efficiency as well as economics of the processing facility. Solids content of LSW was increased from 5 to 16% TS after implementing the RO process; plant operations were stable when LSW concentrations were as high as 20% TS. Membrane flux-

es were 11.5 to 15.8 L/m²/h (LMH) and typically 13.8 LMH. Total nitrogen (TN) concentrations in the permeate were initially 500 to 700 mg/L when membranes were replaced once a year, then decreased to 200 to 300 mg/L when 50% of the membrane area in the RO system was replaced every 6 to 7 months.

Before installation of the RO system, *Kollacks* and *Rekers* [11] reported that the wet mill used 1.5 to 1.6 m³ fresh water per t corn (2.9 L water per kg dry starch) with LSW of 5% TS and a draw rate of 800 to 900 L/t corn. With the RO system, the LSW draw rate was reduced to an average of 300 L water per t corn but reductions as low as 175 L per t were observed. Fresh water consumption dropped to 2.2 L wash water /kg dry starch and steam use dropped to 41 to 46% of use before RO use. The RO system was operated along with a nine stage starch washing system and gave starch quality comparable to a 15 stage wash system without RO. They found that RO of LSW was not an acceptable method to generate water for recycling within the plant; small molecular weight components contained in LSW, such as ethanol, amino acids, lactic acid, would permeate an RO membrane. Small molecular components in the permeate were not desirable for use in the starch washing operation, unless these impurities did not impact the particular starch products being made (such as in an ethanol plant). LSW needed to be concentrated to 50%, which would have very high osmotic pressures and entirely preclude practical operation of an RO system, that eliminated evaporation. Small weight constituents and high osmotic pressure resulted in very low flux rates, severe fouling and low permeate quality. The application of RO to the light middlings stream [11] achieved an increase in LSW solids similar to the increase achieved by applying RO directly to the LSW stream [7, 12]. This research showed how membrane technology could be used to remove the coupling of steepwater evaporation and fresh water used for starch washing. RO of light middlings produced a source of fresh water that could be used internally to wash starch to remove protein as needed, without increasing evaporative load.

Ray et al. [7] used RO as a preconcentration step prior to conventional steepwater evaporation. LSW was concentrated from 6 to 12% TS using a polyethersulfone hollow fiber RO membrane operating at 50 °C and 2700 kPa in a single stage feed and bleed configuration. The hollow fibers had 300 μm inside diameter. The system was able to operate long term (more than 180 days) filtration experiments in a continuous flow mode. Permeate flux rates were reported to be 3 to 9 LMH. LSW used in experiments was reconstituted from heavy steepwater (50% TS) and distilled water; as a result, these flux rates may not represent actual fluxes on LSW taken directly from the steeping process. The permeate stream was reported to have a COD of 7,000 to 9,800 mg/L compared to commercial evaporator condensate having approximately 1,500 mg/L COD. Similar to conclusions reached by *Kollacks* and *Rekers* [11], *Ray* et al. [7] concluded that the RO permeate would not be suitable for starch washing but could be recycled for steepwater use or other locations in the process.

Several approaches to LSW concentration were used by *Ray* et al. [7] to determine the economic feasibility of an RO system: 1) mechanical vapor recompression (MVR) to concentrate LSW to 50% TS, 2) RO filtration prior to MVR, 3) triple effect evaporation, and 4) RO filtration prior to triple effect evaporation. For each scenario, they varied the amount of water removed by the RO system (from 0 to 70% removal) and determined capital, operating and total production costs for concentration of LSW to 50% TS. While triple effect evaporators traditionally have been used in steepwater concentration and are lower in capital cost, they are being replaced by MVR due to higher energy efficiency. *Ray* et al. [7] found that a combination of RO and triple effect evaporation had lower capital cost compared to RO with MVR, regardless of the amount of water removed by RO. However, RO with MVR was lower in total production (capital plus operating) costs. The RO-MVR method had optimum total production costs at 60% water removal by RO. At 60% water removal, LSW having 7% TS would be concentrated to 12.5% TS using RO filtration; MVR would be used to concentrate from 12.5 to 50% TS.

Gienger and *Ray* [12] used data from *Ray* et al. [7] to simulate RO processes in combination with conventional drying operations. They studied economic feasibility of two simulated membrane applications: RO concentration with conventional MVR to concentrate steepwater and an RO-MVR process to recover energy from dryer exhaust gases. In the case of steepwater concentration, steepwater is pumped through an RO system to remove a portion of water prior to being sent to MVR for final concentration to 45 to 50% TS. Steepwater has an osmotic pressure of 1,000 kPa at 6% TS which increases to 2,400 kPa at 15% TS. Operating pressures of the RO system were 3,400 kPa and a pressure differential of 1,000 kPa was needed between osmotic pressure and fluid pressure for flux rates to be effective. LSW solids content of 15% was the maximum considered for optimization calculations. While *Gienger* and *Ray* [12] did not report any membrane filtration data, their calculations indicated when RO was used to remove 57% of water in LSW prior to MVR, the RO-MVR evaporation process was at an optimum in total production costs. For the membrane based system, electrical, thermal and total cost requirements were 30, 40 and 33% of the conventional steepwater concentration process, respectively.

Much of the energy used to dry coproducts in the wet milling process is lost in the form of latent energy by the venting of warm moist air to the atmosphere. Since water vapor contained in the vented air is a source of energy when condensed, MVR could be used to recover this energy and improve efficiency of drying processes. However, the amount of water vapor per unit mass of vented air is relatively low, making MVR ineffective. The second (RO-MVR) membrane application simulated by *Gienger* and *Ray* [12] was membrane filtration of vented air from coproduct dryers prior to compressing the vapor. This was an attempt to make MVR of water vapor more feasible by removing dry air and, in effect, concentrating the amount of water vapor in vented air subject to MVR by using membranes with selective permeability. Vented air from dryers containing water vapor was passed through an RO membrane and was subjected to an MVR process. The system was designed such that the compressor drew vented air through the membrane. Two membranes tested were impermeable to air while being highly permeable to water vapor. The membrane selected played a large role in total costs, since it affected capital investment as well as selectivity to water vapor and thus ability to recover energy in the form of latent heat. Based on their experimental results with these two selective membranes, *Gienger* and *Ray* [12] concluded that 30% of energy originally introduced into a drying operation could be recovered by this method.

Wu [13] investigated UF followed by RO to concentrate steepwater from corn wet milling. The process was based on previous data from RO and UF-RO filtration processes with stillage materials produced from several cereal grain sources [14–21]. *Wu* et al. [21] had found that RO filtration alone resulted in filtration difficulties due to rapid fouling. *Wu* [13] collected samples of commercial light steepwater and used a centrifuge (at 45,200 $\times$ g) to remove suspended solids (7% of steepwater weight) prior to UF. Spiral wound UF polysulfone or cellulose acetate membranes having 1 m^2 area were operated at 680 kPa. The RO system used a polyamide membrane with 1.1 m^2 area that operated at either 5,440 or 6,800 kPa. Permeate flux rates from the UF membrane were 7.6 and 8.6 LMH for polysulfone and cellulose acetate membranes, respectively. Permeate flux rates for most RO experiments were 9.4 and 18.8 LMH at 5,440 and 6,800 kPa, respectively. During filtration, total nitrogen and TS in UF and RO permeate streams tended to increase. UF combined with RO was found to remove over 99.8% of total nitrogen, TS and ash present in the steepwater subjected to centrifugation. Using the higher transmembrane pressure for RO filtration was recommended since it improved permeate flux rates and resulted in a higher level of nitrogen, solids and ash being removed from the permeate.

Singh and *Cheryan* [2] provided a conceptual overview of potential membrane applications in the corn wet milling process, including membrane technology to concentrate LSW and light middlings streams and to facilitate protein extraction of germ and corn gluten meal and corn oil refining. Concentration of LSW by RO was reviewed; ability to concentrate LSW using RO was reported to be a function of osmotic pressure. They reported that practical operating pressures of 4,100 kPa and a minimum driving force of 1,400 kPa would be needed for efficient operation of an RO system, limiting steepwater concentration by RO to 14 to 18% TS. They also described a process that used a combination of MF, UF, and RO to isolate and concentrate proteins in corn gluten meal extracted with alkali and alcohol. In high valued corn gluten meal, proteins originate from the endosperm of the corn kernel, and contain primarily glutelin and zein. Since glutelins are soluble in alkali solutions, and zein is soluble in alcohol solutions, the process takes advantage of preferential solubilities by extraction first in alkali. Glutelin and zein are recovered as UF retentates following their respective extraction in either alkali or alcohol and microfiltration.

Mannheim and *Cheryan* [22] used a combination of enzyme hydrolysis and two stage UF of corn gluten meal to modify and extract proteins. The authors reported functional qualities of proteins recovered, but no flux data or economic feasibility were given. With treatment using protease, proteins from corn gluten meal were converted to soluble peptides having higher solubility and clarity. The treated corn gluten meal was passed through a hollow fiber UF membrane having a 30,000 molecular weight cut off (MWCO). The permeate was passed through a second hollow fiber UF membrane having a 10,000 MWCO. Treatment improved foam forming properties and increased moisture sorption characteristics relative to unmodified corn gluten meal, but these properties and characteristics were not compared to proteins from dairy or soy sources.

Following separation of starch and protein in the primary starch separator, light gluten (LG) is produced which contains 4 to 6% TS (Fig. 1). This process stream is dewatered in the gluten thickener centrifuge to produce heavy gluten (HG); additional water is removed from HG in the rotary vacuum belt filter. Gluten material from the belt filter is dried to allow handling and storage of the resulting corn gluten meal coproduct. Water removed by dewatering is reused elsewhere in the process, and contains 2 to 3% solids which could have been used to increase corn gluten meal yields.

Singh et al. [23] investigated MF to remove water from gluten streams using a stainless steel tubular membrane having 0.1 μm nominal pore size and 0.011 m^2 membrane

area. A batch system was used that operated at 200 kPa transmembrane pressure and 2.5 m/s crossflow velocity to dewater LG (5.2% TS) and HG (13.5% TS). They found average permeate flux rates for LG and HG to be similar, 36 and 34 LMH, respectively. The system was able to achieve solids content of 9.2 and 16.7% for LG and HG samples, respectively; it appeared that concentration to higher solids levels were possible.

Rausch et al. [24] used tubular stainless steel MF membranes with 0.348 m^2 membrane area, 0.1 μm nominal pore size, and 4.6 m/s crossflow velocity operating at an average transmembrane pressure of 200 kPa and arranged in a batch configuration to measure filtration characteristics of corn wet milling coproduct streams, LSW and LG. Filtration of LSW resulted in permeate flux rates of 20 to 40 LMH. MF was able to increase solids content of LSW up to 16% TS; higher levels of solids in the concentrate appeared possible. The filtration resulted in 9 to 11 % TS to pass through as permeate. A preliminary economic analysis was conducted for a wet milling facility processing 2,500 t per day and generating corn gluten meal (CGM) having 62% total protein (wet basis). It was found that up to 48% of LSW volume produced could be concentrated using MF and added to CGM, resulting in a 60% total protein (wet basis) level. With the addition of LSW solids to CGM, yields would increase, add revenue to facility operation and result in a payback period of 1 to 2 years for the MF system. The permeate from MF could be concentrated using conventional evaporation. It was determined that the MF permeate had a fivefold reduction in evaporator fouling tendencies compared to conventional LSW. Filtration experiments with conventional LG measured flux rates of 30 to 50 LMH and concentrated gluten solids levels as high as 16% TS, similar to HG solids content. Permeate TS remained low, at 2 to 3% TS, during all filtration experiments. This work indicated that MF of LSW and LG process streams would be a feasible alternative to conventional evaporation and centrifugation unit operations.

Thompson et al. [25] studied changes in nutrient composition of a commercial gluten stream when MF was used to dewater LG and HG. The membrane was of stainless steel tubular design similar to those used by *Singh* et al. [23] and *Rausch* et al. [24] and operating at an average transmembrane pressure of 200 kPa and a membrane area of 1.74 m^2. Nutrient compositions of LG and HG concentrated using MF were compared to those of conventional methods. The gluten dewatering and drying process was sampled at six locations in a commercial wet mill. Samples included LG, HG, process water from the gluten thickener centrifuge, filtrate from the rotary vacuum belt filter, wet gluten cake and dried CGM (Fig. 1). Membranes were cleaned with a solution containing sodium hydroxide and sodium hypochlorite heated to 90 °C after each run; flux rates were restored to within 90% of original clean water rates. Permeate flux rates for LG and HG were approximately 40 LMH. MF dewatered LG and HG up to 15 and 21% TS, respectively; higher TS levels were limited by capacity of the pump. Nutrient data indicated TS content of permeate streams were a third lower (1.9 to 2.0% TS) compared to conventional process water streams (2.5 and 3.6% TS for gluten thickener overflow and vacuum belt filtrate, respectively) but contained much higher levels of ash (10.0 to 14.5% compared to 6.0 to 6.3%, dry basis).

Simms [26] stated that one of the most common applications for membranes in corn wet milling is removal of residual lipids, proteins and colorants from corn syrup. These residual materials, commonly referred to as "mud", are released from the starch granule during conversion to corn syrup and must be removed through several refining processes. Conventionally, much of the residual material is removed using a rotary vacuum filter coated with diatomaceous earth. As vacuum pulls syrup through the filter, the diatomaceous earth removes residual materials. Eventually, used diatomaceous earth must be removed from the filter and deposited in a landfill or other method of disposal. The vacuum filter must then be recoated, resulting in a high maintenance operation. Use of MF to replace conventional rotary vacuum filtration resulted in syrup with higher clarity, lower operating costs for clarification and elimination of disposal of diatomaceous earth. In a syrup line operating at 2,000 L/min, annual savings over \$ 2×10^6 were reported. *Simms* [26] also reported that in 1997 approximately 75% of U.S. produced corn syrup was clarified using membrane filtration instead of conventional vacuum filtration.

Membrane filtration is also effective in the final filtration of corn syrup products just prior to shipment to users of corn syrups [26]. Many food and beverage manufacturers require assurances that syrup is free of residual particulates and microorganisms. Use of MF or UF with nominal pore sizes of 0.01 to 0.1 μm will provide pasteurization without use of heat, as most microorganisms are larger than this pore size. A typical application would be to filter high fructose corn syrup through a MF membrane with 0.1 μm nominal pore size operating at 1,400 to 4,800 kPa.

Chemically modified starch products are produced by treating starch in single or dual reactions. These reactions require addition of buffering salts to protect granule integrity during modification. Residual modification chemicals, reaction products, and buffering salts must be removed from the starch prior to dewatering and drying. Residual materials in the modified starch slurry are removed using various washing methods, such as centrifu-

gation. Washing losses of 3 to 8% of starch solids are common [26]. Membrane systems can recover modified starch, improving yield and reducing waste treatment costs. Fluxes between 68 and 200 LMH for filtration of starches at solids levels of 25 to 39% TS were reported and were dependent on characteristics of the modified starch. *Simms* [26] reported that stainless steel tubular membranes can dewater starch up to 40% TS, reducing water to be removed by subsequent dewatering and drying steps.

A ceramic multichannel microfiltration membrane was used to clarify a 95 dextrose equivalent (DE) commercial corn starch hydrolysate having 29% solids and pH of 3.3 [27]. Experiments were conducted at a process temperature of 60 °C and for periods of 8 h. The membrane had 0.2 m² surface area and nominal pore size of 0.2 μm. Crossflow velocities were 2 and 5 m/s, and volumetric concentration factors varied between 1 and 100. Conditions for maximum flux rates occurred at transmembrane pressures of 200 to 340 kPa. Flux rates decreased from 145 to 130 LMH with higher volume concentration ratios from 10 to 100.

Filtration data collected with this system were used by *Singh* and *Cheryan* [27] to investigate the economic feasibility of a commercial system clarifying 95 DE hydrolysate flowing at a rate of 114 m³/h. Higher crossflow velocities (5 m/s) through the membranes increased energy costs, but decreased total operating costs since the required membrane area was lower and replacement costs were nearly half the amount at the lower velocity (2 m/s). Total operating costs were found to be 30% of the conventional method of rotary vacuum belt filtration.

3 Potato Starch Processing

Eriksson and *Sivik* [28] stated that process water from manufacture of potato starch (Fig. 2) presents a major disposal challenge. More than 40% of TS in process water consists of protein or other nitrogen containing solids. Potato protein quality is similar to egg protein and higher in value than most other plant proteins [29]. Typical potato process water contains 2.8% protein, 1.4% ash, 1.2% fiber and 1.1% sugar (6.5% TS). These process streams are dilute (2 to 7% TS), and costly to concentrate by evaporation; they also are relatively high strength wastes and are expensive to treat. Production of potato starch results in losses of approximately 20% of potato solids [30]. A primary means of disposal of these solids is through irrigation of crop land with process water. This method of disposal is becoming more restricted due to costs and environmental regulations [31]. Part of the potato protein in process water can be recovered through coagulation using heat; the remainder of solids are recovered through centrifugation and evaporation. Newer technology using decanter type centrifuges in multiple stages and three-phase nozzle separators has reduced water needs for potato starch production [31] but recovery of nutrients in process water remains an issue.

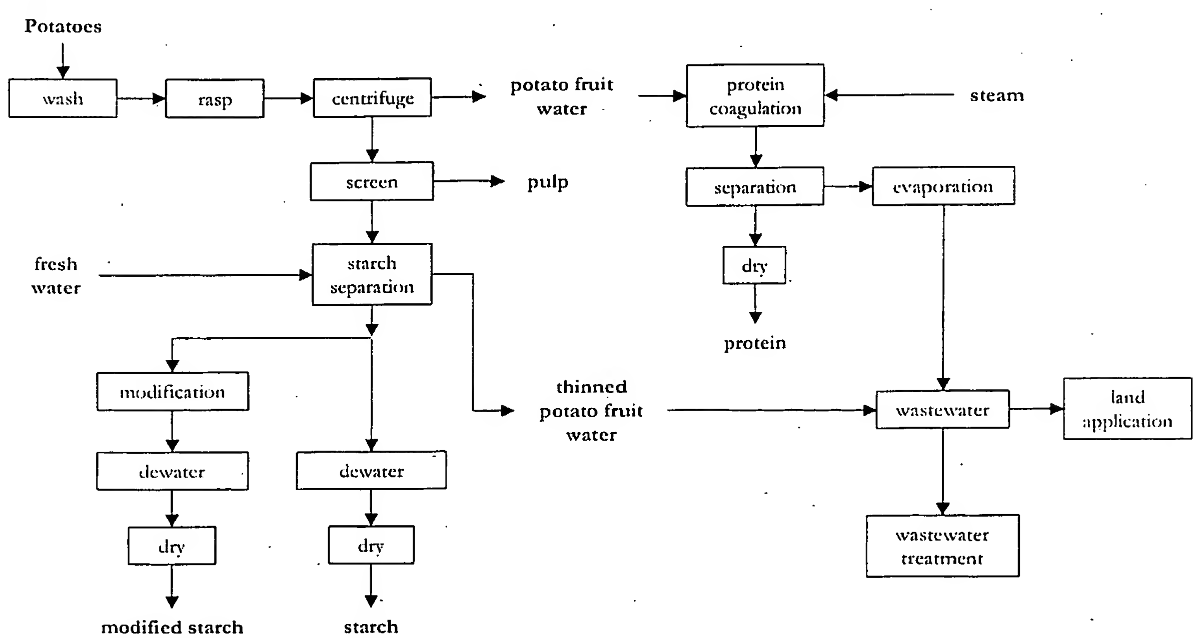

Fig. 2. Schematic of the process to produce potato starch [34].

Eriksson and *Sivik* [28] used tubular cellulose acetate UF membranes having a 30,000 MWCO and operating at 300 to 700 kPa to concentrate potato process water to 18% TS. Flow velocity of 4.2 m/s gave flux rates about 60% higher than at 1.7 m/s. Membranes were more difficult to clean when lower flow velocities were used. *Meindersma* [32] studied concentration of potato process water using a combination of UF and RO filtration techniques. Membrane filtration applied after juice separation and defoaming was studied. Several membrane module configurations were tested for the filtration study, including tubular, flat and spiral wound designs. Flat and spiral wound configurations proved to be problematic with this type of process water due to clogging of modules. Tubular module configurations operating gave the best results, since this configuration was not clogged by process solids. UF concentrated high molecular weight materials, such as proteins, but had 2.5 to 3% solids in permeate; preventing direct discharge of permeate. RO membrane filtration operating at 2,000 to 5,000 kPa reduced COD and nitrogen levels by over 99% in the permeate [32]. Therefore, analysis and evaluation was focused on RO alone. An RO filtration system was found to have a payback period of 3 years when energy savings were considered . Electric energy, cleaning and membrane replacement accounted for 44, 26 and 30%, respectively, of operating costs for the RO concentration system [32].

Meuser and *Köhler* [30] sought membrane technology as a cost saving step prior to both coagulation and evaporation processes. They evaluated UF and RO filtration techniques for solids recovery from potato process water, operating at 4,000 kPa, 25 °C and linear flow velocity within the modules of 2.5 m/s. UF resulted in COD reduction of 90% in potato process water, while RO reduced COD by 99%. Although UF had higher flux rates of 40 LMH, this method of filtration demonstrated no advantages over RO filtration; UF permeate could not be discharged without additional treatment. RO filtration doubled potato juice solids concentration and reduced energy requirements by a third; total costs were reduced by approximately 25%. Both thermal and electrical energy requirements were reduced when using the RO technique.

Boruch et al. [33] compared the ability of an acid-thermal process to that of a UF process to recover and concentrate potato process water. The acid-thermal process involved coagulation with acid, followed by centrifugation to remove precipitate and evaporation of the centrifuge supernatant to recover a powdered product. The UF process included membrane filtration followed by evaporation and drying of UF filtrate to recover product. The UF membrane was a capillary tube configuration made of polyacrylonitrile material arranged in a bundle, each tube having an inner diameter of 1 mm for a total membrane area of 0.027 m^2. Operating conditions included a linear flow rate within capillaries of 0.6 m/s, pressure at the inlet to the module of 200 kPa and a fluid temperature of 20 °C. Energy needed for the acid-thermal process was nearly twice that required for the UF process. However, the acid-thermal process used for comparisons in the study was a simulated one using lab and pilot scale equipment and may have had higher energy usage than a commercial scale process. The protein rich powder obtained with the UF process was reported to be of higher quality than that from the acid-thermal process, due to lower heat energy used to concentrate and dry the UF product.

One challenge facing potato processors is that facilities often process potatoes during fall and winter months, then switch to winter wheat as it becomes available. As a result, a wide range of process waters and wastewaters are created at a single facility; these process waters have a variety of nutrients and membrane filtration characteristics. *Ruffer* et al. [34] studied this issue using a pilot scale RO filtration unit installed at a facility to filter a portion of process water, or potato fruit water (Fig. 2). This process water originated from the overflow of centrifuges used to separate protein and other solubles from potato starch after the rasping operation. The RO membranes were constructed of cellulose acetate and operated with a linear flow velocity of 2.5 m/s and an average pressure of 4,000 kPa, with a pressure drop of 500 kPa from module inlet to outlet. The RO unit had 180 tubular modules and a total area of 456 m^2. Permeate from the RO unit was recycled for use in the starch extraction step, while concentrate was mixed with other process waters prior to a protein coagulation step. Following coagulation, remaining process waters were further concentrated using an evaporation step. *Ruffer* et al. [34] did not investigate an additional membrane filtration step following coagulation, in contrast to earlier work by *Meuser* and *Köhler* [30]. *Ruffer* et al. [34] tested an RO system for three months which had reliable performance. Permeate fluxes observed varied from 10 to 20 LMH during tests. A detailed economic analysis showed the process of RO filtration followed by coagulation and evaporation was more economical than using evaporation alone; the conventional process was approximately three times more expensive than an RO process, due to reduction of evaporator load.

4 Wheat Starch Processing

Meuser and *Smolnik* [35, 36] applied UF and RO membranes to recover solubles from wheat starch process water. They compared costs of evaporating process water to costs for membrane filtration in combination with evaporation and found that UF and RO membranes could be used to filter process water to reduce water pollution and

recover soluble solids. The COD of the permeate was directly proportional to the COD of the retentate stream, but filtration was limited by short membrane life and low permeate flux rates for these membranes. As a result of low permeate flux rates, a large membrane area was required and the process was not economical.

Fane and *Fell* [37] evaluated two common processes used to recover starch and gluten from wheat flour: the Martin process and the batter process. Both processes used water to separate starch from gluten; the Martin process (Fig. 3) agglomerated gluten while the batter process (Fig. 4) dispersed gluten and collected it as a fine curd product [38]. These processes were used to produce vital wheat gluten, a protein concentrate used extensively

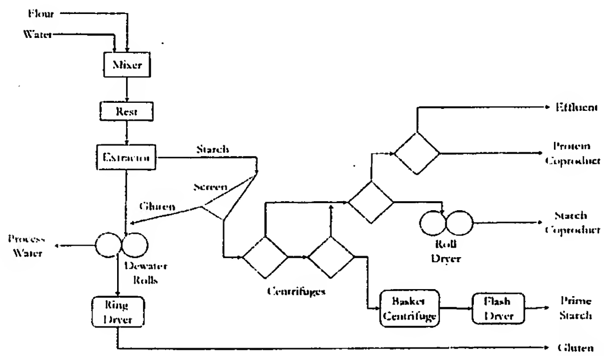

Fig. 3. Schematic of the Martin process for production of wheat gluten [38].

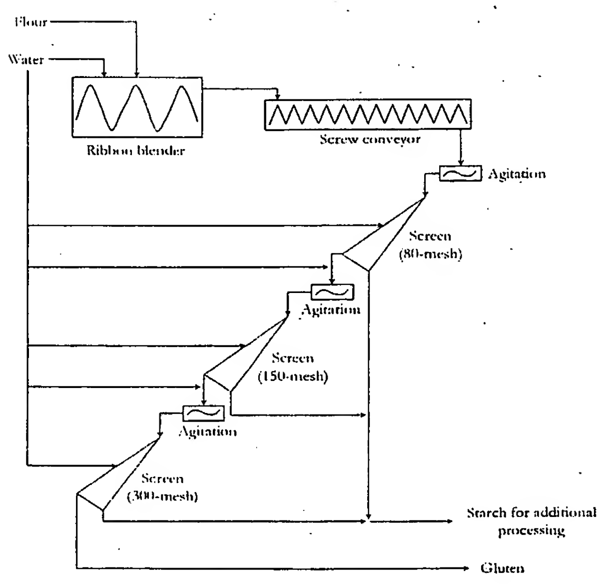

Fig. 4. Schematic of the batter process for gluten production [38].

in baked goods. At the time of their work, both processes required approximately 15 kg of water per kg of flour processed. Wastewater was treated either on site or at a municipal treatment system, or in some cases the waste was applied to land. Over the years, costs of both disposal methods continued to rise, giving processors incentive to investigate ways to reduce both strength and volume of effluent.

Discharge of water was not the only problem associated with processing of flour. *Fane* and *Fell* [37] reported 13% of solids entering a typical wheat flour processing plant were lost in waste effluent. A protein balance indicated 16% of protein entering the facility was lost as waste, representing a loss of potential product. The facility processed 400 t of wheat flour per day and generated over 5,100 t of wastewater. Most proteins found in wastewater were soluble and, if recovered by a membrane process and spray dried, were found to improve baking performance of gluten. Therefore, the large volume of wastewater represented a double cost liability: waste disposal costs and lost revenue due to loss of protein.

Fane and *Fell* [37] carried out an extensive analysis of wheat starch process effluent; typical effluent contained 0.85 to 1.2% TS, had a five day biological oxygen demand (BOD_5) of 4,000 mg/L and pH of 3.4 to 3.8. Effluent solids contained starch and sugars, amino acids, ash and pentosan levels of 65.0, 18.0, 6.3 and 5.1%, respectively. These characteristics created a challenge due to low solids content and pH and high total volume and BOD_5.

Membrane applications had been used for treatment of cheese whey and soy whey in a limited number of treatment plants [37]. For dairy applications, these installations typically used a two step membrane process; UF was used to recover protein and RO was used to recover lactose. A similar pilot scale investigation was conducted on site by *Fane* and *Fell* [37] at a commercial wheat processing facility. Equipment was configured in a recirculating batch loop consisting of a batch tank, pump, heat exchanger and membrane module and operated at approximately 200 to 350 kPa. Several UF membranes ranging in MWCO from 24,000 to 120,000, membrane areas varying from 0.15 to 1.7 m^2 and membranes constructed of cellulose acetate and noncellulosic materials were included in the study. Membranes performed similarly in terms of permeate flux rate when filtering wastewater having 1 to 10% TS. Fluxes typically began at 20 LMH and decreased until steady values near 10 to 12 LMH were reached. Membranes were cleaned effectively using a protease enzyme as part of the cleaning regimen. The permeate contained no microorganisms, but did contain sugars and inorganics. This indicated that UF permeate could potentially be used as a water source within the

process, but tolerance of the process to presence of sugars and organics had not been determined. *Fane* and *Fell* [37] suggested use of RO may be needed if process water was to be extensively recycled. Concentrate from the UF process was spray dried and tested as a gluten substitute in bread baking. Up to 7.5% of gluten could be substituted without any loss in loaf volume or texture. This demonstrated potential for UF concentrate to be recovered and sold, rather than being a disposal cost. However, potential market for the gluten substitute was largely unknown and difficult to factor into an evaluation.

Fane and *Fell* [37] also performed an economic analysis to determine feasibility of a combination UF concentration and spray drying operation to produce an animal feed from recovered solids. Based on protein content, dried solids were valued at $ 130 per t. This study did not investigate other methods of drying UF concentrate. Costs for cleaning membranes were not reported, but the authors noted that annual costs for UF operation were one tenth the cost of the spray dryer. The payback period would be approximately seven years and was highly sensitive to membrane flux rate and maximum achievable retentate concentration. Because the economic return was highly dependent on membrane operating parameters, additional work was recommended.

Harris [39] updated the work of *Fane* and *Fell* [37] by using current membrane materials and economic parameters to evaluate membrane filtration in wheat processing. Three types of membranes were investigated extensively, constructed of either poly(vinylidene fluoride) or polysulfone, with each module having a surface area of 0.84 m^2, MWCO ranging from 50,000 to 100,000 and an average transmembrane pressure of 500 kPa. Linear flow rates through the membrane tubes ranged from 1.38 to 2.76 m/s., to study the effect of the Reynolds number (which is a function of flow velocity and fluid viscosity) ranging from 11,100 to 44,000. Effect of enzyme pretreatment on membrane flux rate was also investigated. The permeate flux rate was found to have an increasing exponential relationship with Reynolds number. The Reynolds number was found to decrease during concentration to a value as low as 390. Concentrate from filtration contained 88% of initial protein; it appeared that some proteins had molecular weights less than 20,000 and would require a membrane of lower MWCO if these proteins were to be recovered. Although treatment of concentrate with a hemicellulase during UF improved permeate flux rates, spray drying UF concentrate to produce an animal feed ingredient (worth $ 400 per t) was not economically feasible. If the dried product could be sold as a breadmaking ingredient valued at $ 800 per t, the economic picture was only marginal, and strongly dependent on membrane service life, protein rejection and hemicellulase concentration. This demonstrated the sensitivity of membrane process feasibility on market value of coproducts. Processors adopting this alternative must have an understanding of market opportunities for the new coproduct. While the returns were not attractive, the economic picture could have changed if wastewater treatment costs increased.

Sutton [40] used a membrane anaerobic fluidized bed reactor treatment system to treat several types of cereal process wastewaters, including that from a plant processing wheat flour. The system used an adaptation of a fixed film biological reactor. Wastewater and effluent recycle were pumped upward through the reactor at a velocity sufficient to suspend growth media. The media were particles that provided a large surface area for biological growth relative to the volume of the reactor. The high surface area provided by the fluidized media greatly reduced hydraulic retention time and reactor size needed to treat wastes. Biomass concentration was five to ten times greater than when maintained in a typical suspended growth system. Following anaerobic stabilization, reactor contents were pumped through a UF membrane. Permeate was discharged from the waste treatment system, while retentate was returned to the reactor. This system was considered cost effective when used with low volume, high strength (COD greater than 15,000 mg/L) wastewater due to the size and cost required for the UF system. *Sutton* [40] used a pilot scale (300 L) reactor system to treat wheat processing wastewater, which consisted primarily of washwater from starch and gluten production. No information was given on the UF membrane used with the reactor. Using the pilot scale system, *Sutton* [40] found COD, BOD_5 and TS reductions of greater than 99% with a loading rate of 8.2 kg COD per m^3 per day. Biogas was also produced with the treatment process and contained 68% methane. Yield of methane was 0.29 m^3 per kg COD.

5 Conclusion

Much has been published to demonstrate effectiveness of membranes in starch processing. There are many potential applications that must be developed individually (maize, potato, wheat starch, etc.), each having several process streams where membranes could be used. Each process has its own unique economic and process variables. In addition, effectiveness of membrane technologies have continued to improve, making membranes more resilient and cost effective.

It seems that two factors must be present for a membrane application in starch processing to be feasible and effective in starch processing: 1) the membrane process must be economical and 2) the membrane process results in improved quality of the products being manufactured.

Several RO processes reported were shown to save money by reducing energy needs for evaporation. They also showed quality of the starch produced with the revised process was equal to the conventional process. Use of MF to clarify dextrose syrups has been shown to save operating costs and disposal difficulties associated with filter aids used with rotary vacuum filtration. Syrup of high clarity and quality was achieved more reliably with MF membrane techniques. These benefits, taken together, encouraged processors to accept the concept and to switch to a membrane process. Clarification of corn syrups is now the largest membrane application in the starch processing industry.

Conservative processors may be more willing to adapt membrane processes in the future, and thus accelerate implementation of membrane technologies for more subtle benefits. The current challenge at hand is to obtain a combination of economic, nutritional, environmental, and product quality data using methods that conclusively determine when a membrane application concept is suitable for commercial use.

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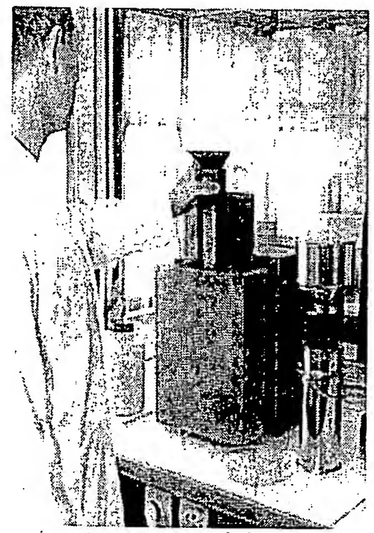
L'USIPA
Un peu d'histoire
Le conseil de direction
Les adhérents
Produits et marchés

L'AMIDONNERIE FECULERIE
L'amidon : un ami naturel
> Les procédés d'extraction
Un secteur dynamique
Une agro-industrie de pointe

LES PRODUITS AMYLACES
Les amidons
Les amidons modifiés
Le glucose
Coproduits et nutrition animale
Les valorisations non-alimentaires

LES PROCEDES D'EXTRACTION

Le métier de l'amidonnerie-féculerie consiste à séparer les constituants de la plante: l'amidon, la protéine, l'enveloppe cellulosique, les fractions solubles et dans le cas du maïs, le germe dont sera extraite l'huile. Cette première phase fait intervenir une série d'étapes simples de séparation physique des constituants: broyage, tamisage, centrifugation etc... A noter toutefois que les procédés de fabrication sont spécifiques à chaque plante et que les outils industriels sont dédiés à une matière première (blé, maïs ou pomme de terre en Europe) sans qu'il y ait donc possibilité de substituer l'une à l'autre. Dans un deuxième temps, l'amidon ainsi extrait sous sa forme la plus pure sera ou bien valorisé "tel quel" après séchage (on parlera "d'amidon natif") ou bien envoyé dans de multiples "ateliers" où il subira diverses transformations visant soit à en améliorer les performances ("amidons modifiés") soit à obtenir les sirops de glucose. Ces derniers donneront à leur tour naissance à de multiples produits finis... mais c'est une autre histoire!


Analyse des céréales

Au final l'amidon et les produits dérivés seront livrés aux industries clientes sous forme de poudre (à l'image de la farine) ou de sirops.

A noter qu'en français, contrairement à l'anglais qui n'utilise qu'un seul terme (starch), l'amidon est appelé "fécule" quand il est extrait des tubercules ou des racines (pommes de terre, manioc ou patates douces).

Translation

Extraction processes

The craft in the starch and potato starch industry consists in separating the plant constituents: starch, proteins, cellulose envelopes, soluble fractions; and for corn, germs, from which oil is extracted. This first phase involves a series of simple steps for physically separating the constituents: grinding, sieving, centrifuging, etc... However, the manufacturing processes are specific for each plant, and the industrial tools are dedicated to one raw material (wheat, corn, or potato in Europe), without it being possible to substitute one with each other. In a second phase, the starch, thus extracted in its purest form, is either used as such after drying ("native starch"), or sent to numerous "workshops", wherein it is processed so that either its performances are improved ("modified starches") or one obtains glucose syrups. The latter in turn lead to multiple finished products... but that is another story!

In the end, starch and derived products are delivered to the client industries in the form of powder (like flour) or in the form of syrups.

In the French language, as opposed to the English language, wherein only one term is used (starch), starch is called "fécule" when it is extracted from tubers or roots (potatoes, manioc, or sweet potatoes). [Translator's note: otherwise, starch is translated as "amidon" in French"]

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